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File Wrapper Information

FULL CONTENTS CLAIM + DETAILED DESCRIPTION
TECHNICAL FIELD PRIOR ART EFFECT OF THE
INVENTION TECHNICAL PROBLEM MEANS
EXAMPLE

[Translation done.]

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Notes:

1. Untranslatable words are replaced with asterisks (*****).
2. Texts in the figures are not translated and shown as it is.

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Dictionary: Last updated 08/17/2009 / Priority: 1. Chemistry / 2.

Mechanical engineering / 3. Technical term

FULL CONTENTS

[Claim(s)]

[Claim 1] In a salt water solution, under existence of an inorganic particle, the monomer 0 of a following general formula (1) - 100-mol %, (Meta) Dispersion liquid which consist of inorganic particle content anionic polymer particulates of 100 micrometers or less produced by making a meltable polymer dispersant live together in this salt water solution, and carrying out the distributed polymerization of 0-100 mol of acrylamide % and this, and the copolymerizable 0-30 mol of nonionic monomer % under churning.

[Chemical formula 1]

[Translation done.]

ID=000003

A general formula (1)

As for SO₃, C₆H₄SO₃, CONHC(CH₃)₂CH₂SO₃, C₆H₄COO, or COO and R₂, in hydrogen, a methyl group or a carboxymethyl group, and A, hydrogen or COOY₂, Y₁, or Y₂ is [R₁] hydrogen or a cation. [Claim 2]Dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1 being more than a kind as which said inorganic particle is chosen from bentonite, kaolin, white carbon, and a talc.

[Claim 3]Dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1, wherein a monomer of said general formula (1) is more than a kind chosen from acrylic acid (meta), itaconic acid, 2-acrylamide 2 **MECHIRU propanesulfonic acid, or these salts.

[Claim 4]Dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1, wherein a meltable polymer dispersant is an ionic polymer into said salt water solution.

[Claim 5]Dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1, wherein a salt which constitutes said salt water solution is a bivalence anion salt.

[Claim 6]In a salt water solution, under existence of an inorganic particle, the monomer 0 of the above-mentioned general formula (1) - 100-mol %, (Meta) A manufacturing method of dispersion liquid which consist of inorganic particle water content solubility polymer particulates of 100 micrometers or less making a meltable polymer dispersant live together and carrying out the distributed polymerization of 0-100 mol of acrylamide % and this, and the copolymerizable 0-30 mol of nonionic monomer % under churning into this salt water solution.

[Claim 7]A manufacturing method of dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 6 being more than a kind as which said inorganic particle is chosen from bentonite, kaolin, white carbon, and a talc.

[Claim 8]A manufacturing method of dispersion liquid which consist of the inorganic particle water content

solubility polymer particulates according to claim 6, wherein a monomer of said general formula (1) is more than a kind chosen from acrylic acid (meta), itaconic acid, 2-acrylamide 2 **MECHIRU propanesulfonic acid, or these salts.

[Claim 9]A manufacturing method of dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 6, wherein a meltable polymer dispersant is an ionic polymer into said salt water solution.

[Claim 10]A manufacturing method of dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 6, wherein a salt which constitutes said salt water solution is a bivalence anion salt.

[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention relates to inorganic particle water content solubility polymer dispersion liquid and a manufacturing method for the same.

It is related with the manufacturing method, concerning the dispersion liquid which consist of inorganic particle content anionic polymer particulates of 100 micrometers or less produced by making a meltable polymer dispersant live together in this salt water solution, and carrying out the distributed polymerization of the specific monomer (mixture) under churning in detail.

[0002]

[Description of the Prior Art]A meltable polymer dispersant is made to live together in this salt water solution in a salt water solution, and the art which carries out a distributed polymerization is indicated by JP,H3-74682,B or JP,H6-72170,B. How to carry out the distributed polymerization of the large drop radial ball child insoluble in water is also known the bottom of inorganic particle existence, and among the salt water solution like JP,H7-88492,A. The method of furthermore carrying out emulsion polymerization under inorganic loading material existence, such as a talc, is indicated to JP,H8-269430,A. However, a water soluble monomer is used as a raw material in a salt

water solution and under inorganic particle existence, and it is not known about the dispersion liquid which consist of a polymer particle with a grain size of 100 micrometers or less which serves as particles without the generated polymer dissolving into a salt water solution, and which carried out the distributed polymerization.

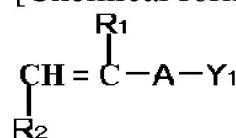
[0003]

[Problem to be solved by the invention]The purpose of this invention is to use a water soluble monomer as a raw material, to examine the dispersion liquid which consist of particles which carried out the dispersion-among salt water solution polymerization under inorganic particle existence about the distributed polymerization which serves as particles without the generated polymer dissolving into a salt water solution, and to develop the manufacturing method and its use further.

[0004]

[Means for solving problem]As a result of examining the above-mentioned technical problem wholeheartedly, an invention which is described below was reached. The invention of Claim 1 of this invention under existence of an inorganic particle in a salt water solution Namely, the monomer 5 of a following general formula (1) - 100-mol %, And it is related with the dispersion liquid which consist of inorganic particle water content solubility polymer particulates of 100 micrometers or less produced by making a meltable polymer dispersant live together in this salt water solution, and carrying out the distributed polymerization of this and the copolymerizable 0-95 mol of nonionic monomer % under churning.

[Chemical formula 1]



General formula (1)

As for SO₃, C₆H₄SO₃, CONHC(CH₃)₂CH₂SO₃, C₆H₄COO, or COO and R₂, in hydrogen, a methyl group or a carboxymethyl group, and A, hydrogen or COOY₂, Y₁, or Y₂ is [R₁] hydrogen or a cation. [0005]The invention of Claim 2 is dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1 being more than a kind as which said inorganic particle is chosen from bentonite, kaolin, white

carbon, and a talc.

[0006]The monomer of said general formula (1) the invention of Claim 3 Acrylic acid (meta), It is dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1 being more than a kind chosen from itaconic acid, 2-acrylamide 2 **MECHIRU propanesulfonic acid, or these salts.

[0007]Dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1, wherein a polymer dispersant with an invention of Claim 4 melttable in said salt water solution is an ionic polymer.

[0008]The invention of Claim 5 is dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 1, wherein the salt which constitutes said salt water solution is a bivalence anion salt.

[0009]The invention of Claim 6 under existence of an inorganic particle in a salt water solution The monomer 5 of the above-mentioned general formula (1) - 100-mol %, And a manufacturing method of the dispersion liquid which consist of inorganic particle water content solubility polymer particulates of 100 micrometers or less making a melttable polymer dispersant live together and carrying out the distributed polymerization of this and the copolymerizable 0-95 mol of nonionic monomer % under churning into this salt water solution.

[0010]The invention of Claim 7 is a manufacturing method of the dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 6 being more than a kind as which said inorganic particle is chosen from bentonite, kaolin white carbon, and a talc.

[0011]The monomer of said general formula (1) the invention of Claim 8 Acrylic acid (meta), It is a manufacturing method of the dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 6 being more than a kind chosen from itaconic acid, 2-acrylamide 2 **MECHIRU propanesulfonic acid, or these salts.

[0012]A manufacturing method of the dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 6,

wherein a polymer dispersant with the Claim 9 invention meltable in said salt water solution of ** is an ionic polymer.

[0013]The invention of Claim 10 is a manufacturing method of the dispersion liquid which consist of the inorganic particle water content solubility polymer particulates according to claim 6, wherein the salt which constitutes said salt water solution is a bivalence anion salt.

[0014]

[Mode for carrying out the invention]A manufacturing method is explained concretely below. The inorganic particle water content solubility polymer dispersion liquid of this invention prepare the aqueous solution of a multivalent anion salt like ammonium sulfate, Into this, an inorganic particle, a water-soluble anionic monomer, nonionic monomers, or those mixtures are carried out, and it is crowded, and a meltable polymer dispersant is made to live together in this salt water solution as a dispersant, and a distributed polymerization can be carried out under churning and it can compound.

[0015]If the inorganic particle used by this invention is an inorganic particle which is not dissolved in acid aqueous solutions, such as bentonite, a talc, clay, kaolin, and white carbon, it can be used, but bentonite, kaolin, and a talc are more preferred. As a grain size, 0.001-0.5 micro is the particles of 0.01-0.2 preferably. Since such a grain is usually mutually condensed as an aggregated particle or Miyoshi grains, it is good to use it, making it swell strong-agitating with a homogenizer or agitating for 12 hours - a little less than 24 hours, before using it for a polymerization.

Although the blending ratio of the inorganic particle to a monomer is one to 300 mass %, it is five to 50 mass % preferably [it is desirable and] to five to 100 mass %, and a pan.

[0016]Next, the character of dispersion liquid is explained. [the dispersion liquid which consist of inorganic particle water content solubility polymer particulates of this invention] Can compound a nonionic water solubility polymer or an anionic polymer, and with a manufacturing method further Dispersion liquid with a polymer melt able in water, There are two kinds of things of the description which will be dissolved if a polymer maintains the state of dispersion liquid only by dissolving and diluting, neutralizes

by an alkali and makes pH seven or more even if it mixes with water. The existing dispersion liquid of the former solubility neutralize 10-20-mol% of the anionic monomers used as a raw material, are made to live together as a polymer dispersant, and can be manufactured by polymerizing agitating.

[0017]On the other hand, only by diluting, use the type which maintains a dispersion state by un-neutralizing, and it makes a polymer dispersant live together, and in the same saline as the above, raw material (meta) acrylic acid polymerizes under churning, and it can manufacture it.

[0018]Next, a dispersant is explained. As a polymer dispersant, although either nonionic or an ionic polymer is usable, the ionic polymer is more preferred and anionic is still more preferred than cationicity. As an example of an anionic polymer, it is a polymer (**) of anionic monomers, such as acrylic acid (meta), maleic acid, itaconic acid, acrylamide 2-methylpropanesulfonic acid (salt), and styrene sulfonic acid (salt). Acrylamide, N-vinylformamide which are further nonionic monomers, A copolymer with N-vinylacetamide, N-vinyl-pyrrolidone, N, and N-dimethylacrylamide, acrylonitrile, diacetone acrylamide, 2-hydroxyethyl (meta) acrylate, etc. is also usable. In addition, they are anion denaturation polyvinyl alcohol, styrene / maleic anhydride copolymer, butene / maleic anhydride copolymer, or those partial amidation things. The most desirable combination is acrylic acid (meta) and 2-acrylamide 2-methylpropanesulfonic acid.

[0019]As a cationic polymer, an acrylic cationic monomer. For example, they are a salt of inorganic acid, such as acrylic acid (meta) dimethylaminoethyl and dimethylaminopropyl (meta) acrylamide, or an organic acid, or a copolymer of quarternary ammonium salt and acrylamide by methyl chloride or benzyl chloride. For example (meta), an acryloyloxyethyl trimethylammonium chloride, A (meth)acryloyloxy 2-hydroxypropyl trimethylammonium chloride, (Meta) An acryloyl aminopropyl trimethylammonium chloride, (Meta) An acryloyloxyethyl dimethylbenzyl ammonium chloride, A (meth)acryloyloxy 2-hydroxypropyl dimethylbenzyl ammonium chloride, an acryloyl (meta) aminopropyl dimethylbenzyl ammonium chloride, etc. may be raised, and the copolymer of these monomers and a nonionic monomer

may be sufficient. Diarylamine system polymers, such as a dimethyldiaryl ammonium salt ghost polymer, can also be used.

[0020]As an example of a nonionic polymer, it is a full amidation thing of the polymer (**) of the above-mentioned nonionic monomer, polyvinyl alcohol, styrene / maleic anhydride copolymer, or butene / maleic anhydride copolymer, etc.

[0021]as the molecular weight of the above-mentioned ionic polymer -- 5 and 000 to 3 million -- it is 50,000 to 1,500,000 preferably. As molecular weight for a nonionic polymer, it is 1,000-1 million and is 1,000-500,000 preferably. The additions to the monomer of these polymers dispersant are 1 / 100 - 1/10, and are 2 / 100 - 5/100 preferably.

[0022]The temperature at the time of a polymerization is 5-50 **, and is 15-40 ** preferably. When it is made higher than 50 **, control of a polymerization is difficult, a rapid rise in heat, agglomeration of polymerization liquid, etc. occur, and stable dispersion liquid are not generated by high polymerization.

[0023]A polymerization start uses a radical polymerization initiator. Either oil-soluble or water-soluble are OK as these initiators, and it is possible for azo, a peroxide system, or a redox system to polymerize. As an example of an oil-soluble azo initiator, 2, 2'-azobisisobutyronitrile, 1, 1'-azobis (cyclohexanecarbonitrile), 2, 2'-azobis (2-methylbutyronitrile), 2, 2'-azobis (2-methylpropionate), 4, and 4-azobis (4-methoxy- 2, 4 dimethyl) valeronitrile etc. are raised, and it dissolves in a **** solubility solvent and adds.

[0024]As an example of a water-soluble azo initiator, it is 2, 2'-azobis (amidinopropane) dihydrochloride, 2, and 2'-azobis. [2-(5-methyl-2-imidazoline 2-yl) propane] Dihydrochloride, 4, 4'-azobis (4-cyano valeric acid), etc. are raised. As an example of a redox system, combination with PERUOKUSO disulfuric acid ammonium, sodium sulfite, sodium hydrogensulfite, trimethylamine, tetramethylethylenediamine, etc. is raised. Furthermore as an example of peroxide, PERUOKUSO disulfuric acid ammonium or potassium, Hydrogen peroxide, benzoIRUPERU oxide, a lauroyl PERUOKI side, octano IRUPERU oxide, a SAKUSHINIKKUPERUOKI side, t-butylperoxy-2-ethylhexanoate, etc. can be raised. 2 these

most desirable initiators of whose are water-soluble azo initiators, 2'-azobis (amidinopropane) dihydrochloride, 2, and 2'-azobis [2-(5-methyl-2-imidazoline 2-yl) propane] It is dihydrochloride.

[0025]When polymerizing a type which dissolves only by diluting especially in water, it is effective if an initiator of a REDOKUSSU system is used. An oxidizing substance and a reducing substance are combined as a redox system initiator. As an example of an oxidizing substance, are

PERUOKUSO disulfuric acid ammonium, PERUOKUSO potassium disulfate, hydrogen peroxide, etc., and, [as an example of a reducing substance] Although it is sodium sulfite, sodium hydrogensulfite, ferrous sulfate, sodium thiosulfate, sodium oxalate, triethanolamine, or tetramethylethylenediamine, combination of PERUOKUSONI ammonium sulfate and sodium hydrogensulfite is the most preferred. Thus, polymer dispersion liquid which controlled a rate of polymerization and were stabilized in high polymerization can be comparatively manufactured by pressing down an addition level of an initiator low at low temperature.

[0026]Sulfone group of a carboxyl group is also usable and an anionic monomer to be used may copolymerize both. An example of a sulfone group content monomer is vinylsulfonic acid, vinylbenzene sulfonic acid, or 2-acrylamide 2-methylpropanesulfonic acid. An example of a carboxyl group containing monomer is methacrylic acid, acrylic acid, itaconic acid, maleic acid, or p-carboxystyrene. Furthermore, a copolymer with other nonionic monomers may be sufficient as these polymer dispersion liquid. For example (meta), acrylamide, N,N-dimethylacrylamide, Vinyl acetate, acrylonitrile, methyl acrylate, and 2-hydroxyethyl acrylate (meta), Diacetone acrylamide, N-vinyl pyrrolidone, N-vinylformamide, N-vinylacetamide, acryloyl morpholine, etc. are raised, and copolymerization with these kinds or two sorts or more is possible. As most desirable combination, they are 2-acrylamide 2-methylpropanesulfonic acid, acrylic acid, and krill amide.

[0027]A copolymerization molar ratio of an anionic monomer is 5-100-mol %, and is 10-100-mol % preferably. A still more nearly copolymerizable nonionic monomer can also be copolymerized. As the quantity, it is 0-95-mol %, and is 0-90-mol % preferably. using the amount of single

which made acrylamide a subject, when manufacturing a nonionic polymer -- said nonionic monomer -- 0-30-mol % -- it may copolymerize.

[0028] Although it is a salt with alkali metal ion, the ammonium ion and halide ion like sodium or potassium, sulfate ion, nitrate ion, phosphoric acid ion, etc. as salts to be used, a salt with a multivalent anion is more preferred. As salt concentration of these salts, it can be used to 7 weight % - saturated concentration. It thinks.

[0029] In the type which dissolves in water the dispersion liquid which consist of inorganic particle content anionic polymer particulates of this invention, it adds as a dilution water solution, and, in a type insoluble only by diluting with water, adds in the wastewater made into the purpose with the form of dilution dispersion liquid, respectively, or a paper-making raw material.

[0030]

[Working example] Hereafter, although an embodiment and a comparative example explain this invention in more detail, this invention is not restrained by the following embodiments unless the gist is exceeded.

[0031]

[Work example 1] An agitator, a flowing-back cooling pipe, a thermometer, and a nitrogen introducing pipe to 500 ml of 4 mouth separable flask which it had Deionized water:65.0g, 60% acrylic acid: 50.0 g and 50% acrylamide:140.3g were added, and 30weight % of sodium hydroxide [8.3 g of] neutralized 15-mol% of the amount of anionic single. 66.7 g (20 for monomer mass %) of bentonite swollen overnight while weak-agitating with dispersion liquid 30 more%, 25.0 g (5.0 for monomer mass %) of acrylamide 2-methylpropanesulfonic acid polymers (molecular weight: 200,000 or 20 Eq % neutralized substance) of the ammonium sulfate 135.4g and a 20 mass % aqueous solution were added. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. 2.5g (opposite monomer, 40 ppm) addition of the 0.2 mass % aqueous solution of PERUOKISONI ammonium sulfate of 0.2 mass % and ammonium hydrogen sulfite was carried out in this order, respectively, and the polymerization was made to start 30 minutes after nitrogen introduction. 2 hours after

initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This trial product is set to trial production-1. The molar ratio of the acrylic acid/acrylamide of this trial production-1 was 30/70, and viscosity was 610 mPa-s. It became clear that it was a 2-20-micrometer grain as a result of microscopic observations. After three months went through these dispersion liquid, when it observed, precipitate of an inorganic substance was not seen but was maintaining the stable dispersion state. A result is shown in Table 1.

[0032]

[Embodiments 2-3] The trial productions 2-3 which consist of acrylic acid/acrylamide =30/70, and bentonite versus monomer 40 mass % and 100 mass % by the same operation as Embodiment 1 were compounded. A result is shown table 1.

[0033]

[Work example 4] 60% acrylic acid:100.2g and 50% acrylamide:80.5g were added to 500 ml of 4 mouth separable flask provided with the agitator, the flowing-back cooling pipe, the thermometer, and the nitrogen introducing pipe, and 30weight % of sodium hydroxide [18.5 g of] neutralized 10-mol% of the amount of anionic single. 133.0 g (40 for monomer mass %) of bentonite swollen overnight while weak-agitating with dispersion liquid 30 more%, 25.0 g (5.0 for monomer mass %) of acrylamide 2-methylpropanesulfonic acid polymers (molecular weight: 200,000 or 20 Eq % neutralized substance) of the ammonium sulfate 135.4g and a 20 mass % aqueous solution were added. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. After [of nitrogen introduction] 30 minutes, 2 and 2'-azobis [2-(5-methyl-2-imidazoline 2-yl) propane] 0.8g (opposite monomer, 400 ppm) addition of the 5 mass % aqueous solution of dihydrochloride was carried out in this order, respectively, and the polymerization was made to start. 2

hours after initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This trial product is set to trial production-4. The molar ratio of the acrylic acid/acrylamide of this trial production-4 was 60/40, and viscosity was 450 mPa-s. It became clear that it was a 2-20-micrometer grain as a result of microscopic observations. In observation three months after, precipitate of an inorganic substance was not seen but was stable. A result is shown in Table 1.

[0034]

[Work example 5] An agitator, a flowing-back cooling pipe, a thermometer, and a nitrogen introducing pipe to 500 ml of 4 mouth separable flask which it had The deionized water 16.0g, 60% acrylic acid: 32.0 g, the itaconic acid powder 14.6g, and 50% acrylamide:132.2g were added, and 30weight % of sodium hydroxide [5.4 g of] neutralized 10-mol% of the amount of anionic single. 133.0 g (40 for monomer mass %) of kaolin homogenized for 10 minutes in 3000 rotations / second with dispersion liquid 30 more%, 25.0 g (5.0 for monomer mass %) of acrylamide 2-methylpropanesulfonic acid polymers (molecular weight: 200,000 or 20 Eq % neutralized substance) of the ammonium sulfate 135.4g and a 20 mass % aqueous solution were added. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. After [of nitrogen introduction] 30 minutes, 2 and 2'-azobis [2-(5-methyl-2-imidazoline 2-yl) propane] 0.8g (opposite monomer, 400 ppm) addition of the 5 mass % aqueous solution of dihydrochloride was carried out in this order, respectively, and the polymerization was made to start. 2 hours after initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was

made to continue for further 15 hours, and the reaction was ended. This trial product is set to trial production-5. The molar ratios of the acrylic acid / itaconic acid / acrylamide of this trial production-5 were 20/10/70, and viscosity was 320 mPa-s. It became clear that it was a 2-20-micrometer grain as a result of microscopic observations. In observation three months after, precipitate of an inorganic substance was not seen but was stable. A result is shown in Table 1.

[0035]

[Work example 6] An agitator, a flowing-back cooling pipe, a thermometer, and a nitrogen introducing pipe to 500 ml of 4 mouth separable flask which it had The deionized water 72.5.0g, 60% acrylic acid: 61.6 g, the acrylamide 2-methylpropanesulfonic acid powder 11.8g, and 50% acrylamide:81.0g were added, and 30weight % of sodium hydroxide [7.6 g of] neutralized 10-mol% of the amount of anionic single. The talc 100.2g (30 for monomer mass %) homogenized for 10 minutes in 3000 rotations / second with dispersion liquid 30 more%, 25.0 g (5.0 for monomer mass %) of acrylamide 2-methylpropanesulfonic acid polymers (molecular weight: 200,000 or 20 Eq % neutralized substance) of the ammonium sulfate 135.4g and a 20 mass % aqueous solution were added. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. After [of nitrogen introduction] 30 minutes, 2 and 2'-azobis [2-(5-methyl-2-imidazoline 2-yl) propane] 0.8g (opposite monomer, 400 ppm) addition of the 5 mass % aqueous solution of dihydrochloride was carried out in this order, respectively, and the polymerization was made to start. 2 hours after initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This trial product is set to trial production-6. The molar ratios of the acrylic acid / acrylamide 2-methylpropanesulfonic acid / acrylamide of this trial production-6 were 45/5/50, and viscosity was 850 mPa-s. It became clear that it was a 2-20-micrometer grain as a result

of microscopic observations. In observation three months after, precipitate of an inorganic substance was not seen but was stable. A result is shown in Table 1.

[0036]

[Work example 7]An agitator, a flowing-back cooling pipe, a thermometer, and a nitrogen introducing pipe to 500 ml of 4 mouth separable flask which it had Deionized water:78.3g, 60% acrylic acid : 50.0 g and 50% acrylamide:140.3g are added, 66.7 g (20 for monomer mass %) of bentonite swollen overnight while weak-agitating with dispersion liquid 30 more%, 20.0 g (4.0 for monomer mass %) of dimethyldiaryl ammonium chloride polymers (molecular weight: 100,000) of the ammonium sulfate 135.4g and a 20 mass % aqueous solution were added. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. 2.5g (opposite monomer, 40 ppm) addition of the 0.2 mass % aqueous solution of PERUOKISONI ammonium sulfate of 0.2 mass % and ammonium hydrogen sulfite was carried out in this order, respectively, and the polymerization was made to start 30 minutes after nitrogen introduction. 2 hours after initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This trial product is set to trial production-7. The molar ratio of the acrylic acid/acrylamide of this trial production-7 was 30/70, and viscosity was 330 mPa-s. It became clear that it was a 2-30-micrometer grain as a result of microscopic observations. In observation three months after, precipitate of an inorganic substance was not seen but was stable. A result is shown in Table 1.

[0037]

[Work example 8]An agitator, a flowing-back cooling pipe, a thermometer, and a nitrogen introducing pipe to 500 ml of 4 mouth separable flask which it had Deionized water:65.8g, 60% acrylic acid : 50.0 g and 50% acrylamide:140.3g are added, 32.5 g (6.5 for monomer mass %) of N-vinyl-pyrrolidone polymers (molecular weight: 50,000) of 66.7 g

(20 for monomer mass %) of bentonite and the ammonium sulfate 135.4g which were swollen overnight, and a 20 mass % aqueous solution were added weak-agitating with dispersion liquid 30 more%. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. After [of nitrogen introduction] 30 minutes, 2 and 2'-azobis [2-(5-methyl-2-imidazoline 2-yl) propane] Dihydrochloride did 5 mass % solution 0.8g (opposite monomer, 400 ppm) addition of, and the polymerization was made to start. 2 hours after initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This trial product is set to trial production-8. The molar ratio of the acrylic acid/acrylamide of this trial production-8 was 30/70, and viscosity was 890 mPa-s. It became clear that it was a 2-30-micrometer grain as a result of microscopic observations. In observation three months after, precipitate of an inorganic substance was not seen but was stable. A result is shown in Table 1.

[0038]

[Work example 9]The ammonium sulfate 125.0g, the sodium sulfate 20.4g, and 50% acrylamide:200.0g were made 500 ml of 4 mouth separable flask provided with the agitator, the flowing-back cooling pipe, the thermometer, and the nitrogen introducing pipe. 66.7 g (20 for monomer mass %) of bentonite and 27.5g (5.5% of opposite monomer) of acryloyloxyethyl trimethylammonium chloride polymer aqueous solution (20% aqueous solution, molecular weight 1,200,000) ***** which were swollen overnight were added weak-agitating with dispersion liquid 30%. Then, ***** (ing) and agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. 3.1g (opposite monomer, 25 ppm) addition of the 0.1 weight % aqueous solution of 0.1weight % of PERUOKUSO disulfuric acid ammonium and ammonium hydrogen sulfite

was carried out in this order, respectively, and the polymerization was made to start 30 minutes after nitrogen introduction. 3 hours after the polymerization start, although it was observed that reactant liquid thickens slightly, a particulate material began to deposit. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This trial product is set to trial production-9. The dispersion-liquid viscosity of this trial production-9 was 520 mPa·s. It became clear that it was a 2-20-micrometer grain as a result of microscopic observations. In observation three months after, precipitate of an inorganic substance was not seen but was stable. A result is shown in Table 1.

[0039]

[Comparative example 1] An agitator, a flowing-back cooling pipe, a thermometer, and a nitrogen introducing pipe to 500 ml of 4 mouth separable flask which it had Deionized water:131.7g, 60% acrylic acid: 50.0 g and 50% acrylamide:140.3g were added, and 30weight % of sodium hydroxide [8.3 g of] neutralized 15-mol% of the amount of anionic single. 25.0 g (5.0 for monomer mass %) of acrylamide 2-methylpropanesulfonic acid polymers (molecular weight: 200,000 or 20 Eq % neutralized substance) of the 20 mass % aqueous solution were added. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. 2.5g (opposite monomer, 40 ppm) addition of the 0.2 mass % aqueous solution of PERUOKISONI ammonium sulfate of 0.2 mass % and ammonium hydrogen sulfite was carried out in this order, respectively, and the polymerization was made to start 30 minutes after nitrogen introduction. 2 hours after initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This trial product is set to comparison-1. The molar ratio of the acrylic acid/

acrylamide of this comparison-1 was 30/70, and viscosity was 470 mPa-s. It became clear that it was a 2-20-micrometer grain as a result of microscopic observations. In order to make it correspond with trial production-1 of Embodiment 1, the monomer for preparation, and when it did nature [of 20] % addition of and stability was observed, precipitate of an inorganic substance was accepted in the bentonite dispersion liquid used for comparison-2 by embodiment-1 on the 1st day of preservation. The result of physical properties is shown in Table 1.

[0040]

[Comparative example 2] An agitator, a flowing-back cooling pipe, a thermometer, and a nitrogen introducing pipe to 500 ml of 4 mouth separable flask which it had Deionized water:131.7g, 60% acrylic acid: 50.0 g and 50% acrylamide:140.3g were added, and 32.5 g (6.5 for monomer mass %) of N-vinyl-pyrrolidone polymers (molecular weight: 50,000) of the ammonium sulfate 135.4g and a 20 mass % aqueous solution were added. Then, agitating, nitrogen is introduced and dissolved oxygen is removed from a nitrogen introducing pipe. A constant temperature bath adjusts internal temperature at 25 ** in the meantime. After [of nitrogen introduction] 30 minutes, 2 and 2'-azobis [2-(5-methyl-2-imidazoline 2-yl) propane] Dihydrochloride did 5 mass % solution 0.8g (opposite monomer, 400 ppm) addition of, and the polymerization was made to start. 2 hours after initiator addition, although the viscosity of reactant liquid rose a little, it did not increase more than it, but polymer particles began to deposit to *****, and viscosity also decreased. Added said initiator in equivalent amount in the place since which 8 hour has passed after a polymerization start, respectively, the polymerization was made to continue for further 15 hours, and the reaction was ended. This comparison article is set to comparison-2. The molar ratio of the acrylic acid/acrylamide of this comparison-2 was 30/70, and viscosity was 600 mPa-s. It became clear that it was a 2-30-micrometer grain as a result of microscopic observations. As a result of doing 20 mass [for a monomer] % addition of glue stock TONAITO like comparative example-1 and observing stability, precipitate of an inorganic substance was accepted in preservation one day. The result of physical properties is shown in Table 1.

[0041]

[Table 1]

実施例	試料名	AAC	IA	AMS	AAM	無機物 a)20 b)40 c)30	分散液 粘度 610 700 1100 450 320 850 330 890 520 470 600
1	試作-1	30			70	a)20	610
2	試作-2	30			70	a)40	700
3	試作-3	30			70	a)100	1100
4	試作-4	60			40	a)40	450
5	試作-5	20	10		70	b)40	320
6	試作-6	45		5	50	c)30	850
7	試作-7	30			70	a)20	330
8	試作-8	30			70	a)20	890
9	試作-9				100	—	520
比較例							
1	比較-1	30			70	—	470
2	比較-2	30			70	—	600

AAC: Acrylic acid, IA:itaconic acid, AMS:acrylamide 2

**MECHIRU propanesulfonic acid, AAM:acrylamide,
dispersion-liquid viscosity:mPa-s, and a molecular weight:
unit are 10,000, a bentonite, b kaolin, and c talc. [0042]

[Effect of the Invention]As shown by the embodiment, even if it saves this invention article several months as compared with the blend article of an inorganic particle and an anionic polymer, precipitate of an inorganic substance is not observed. That is, it is guessed that most portion of an anionic polymer is carrying out graft polymerization of this from the surface of an inorganic particle in order to polymerize under existence of an inorganic particle.

[Translation done.]

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[Japanese \(whole document in PDF\)](#)